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(54) SILICATE LUMINESCENT MATERIALS DOPED WITH METAL NANO PARTICLES AND PREPARATION METHODS THEREFOR

(57) The invention belongs to the field of luminescent materials. Disclosed are silicate luminescent materials doped with metal nano particles and preparation methods there for. The silicate luminescent materials doped with metal nano particles are represented by the chemical formula:$MLn_{1-x}SiO_4$:xRE,yA; wherein M is one or two elements selected from Li, Na and K; Ln is one or two elements selected from Y, Sc, La and Lu; A is a metal nano particle selected from Ag, Au, Pt, Pd and Cu; RE is one or two ions selected from Eu, Gd, Tb, Tm, Sm, Ce and Dy; $0<x\leq0.1$; $0<y\leq0.005$. When silicate luminescent materials doped with metal nano particles of the invention are excited by electron beam, they have higher luminescent efficiency. The luminescent materials are good to be used in field emission light source devices.

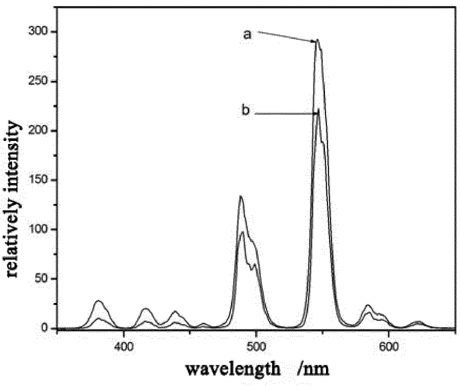

Fig. 2

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Description

FIELD OF THE INVENTION

[0001] The invention relates to the field of luminescent materials, particularly to silicate luminescent materials doped with metal nano particles and preparation methods there for.

BACKGROUND OF THE INVENTION

[0002] In the 1960s, Ken Shoulder proposed ideas based on field emission arrays (FEAs) electron beam microelectronic devices, thus, to design and produce panel display and light resource devices by using FEAs has drawn the public's attention. Similar to the working principle of conventional cathode-ray tube (CRT), such new field emission display lights and forms images by electron beam bombarding on red, green blue trichromatic luminescent powders. Field emission display has potential advantages in luminance, visual angle, response time, working temperature range, power consumption and other aspects.

[0003] A key factor to preparation of field emission display of high performances is to prepare luminescent material of excellent performance. At present, luminescent materials used in field emission devices are sulfide series, oxide series and oxysulfide series luminescent powders. As for sulfide series and oxysulfide series luminescent powders, they have relatively high luminance and electrical conductivity, but, under the large electron beam bombardment, they prone to decomposing into sulfur, which can poison the tip of cathode and produce other precipitates covering the luminescent powders, thus reducing the luminous efficiency of luminescent powders. Oxide and silicate luminescent powders have good stability but the luminous efficiency is not high enough yet.

SUMMARY OF THE INVENTION

[0004] One problem to be solved by the present invention is to provide silicate luminescent materials doped with metal nano particles that can improve luminescent efficiency of field emission device.

[0005] The technical solution of the present invention will be described below.

[0006] A silicate luminescent material doped with metal nano particles represented by the chemical formula: $MLn_{1-x}SiO_4$:xRE,yA; wherein M is one or two elements selected from Li, Na and K; Ln is one or two elements selected from Y, Sc, La and Lu; A is a metal nano particle selected from Ag, Au, Pt, Pd and Cu; RE is one or two ions selected from Eu, Gd, Tb, Tm, Sm, Ce and Dy; x and y are stoichiometric numbers, $0<x\leq 0.1$; $0<y\leq 0.005$. Preferably, x is in a range of $0.001\leq x\leq 0.04$, y is in a range of $0.00003\leq y\leq 0.003$.

[0007] Another problem to be solved by the present invention is to provide method for preparing the silicate luminescent material doped with metal nano particles, comprising:

[0008] S1. According to the stoichiometric ratio of corresponding elements in the chemical formula of $MLn_{1-x}SiO_4$:xRE,yA, weighing compound containing M, compound containing Ln, compound containing RE and silica aerogel containing A ,then grinding and mixing to obtain mixed powders;

[0009] S2, in air or in reducing atmosphere, calcining the mixed powders obtained from S1 at a constant temperature ranged from 800°C to 1600°C for 1h to 10 h; then cooling to room temperature, taking out the calcined matter and grinding to obtain silicate luminescent material doped with metal nano particles represented by the chemical formula of $MLn_{1-x}SiO_4$:xRE,yA, wherein the reducing atmosphere is mixed gases of N_2 and H_2 in a volume ratio of 95:5.

[0010] M is one or two elements selected from Li, Na and K; Ln is one or two elements selected from Y, Se, La and Lu; A is a metal nano particle selected from Ag, Au, Pt, Pd and Cu; RE is one or two ions selected from Eu, Gd, Tb, Tm, Sm, Ce and Dy; x and y are stoichiometric numbers, $0<x\leq 0.1$; $0<y\leq 0.005$.

[0011] Preferably, A is selected from Ag, Au, Pt, Pd and Cu; RE is one ion selected from Eu, Gd, Tb, Tm, Sm, Ce and Dy.

[0012] In S1 of the above preparation method, the compound containing M is selected from oxide of M, nitrate of M, carbonate of M and oxalate of M; the compound containing Ln is selected from oxide of Ln, nitrate of Ln, carbonate of Ln and oxalate of Ln; the compound containing RE is selected from oxide of RE, nitrate of RE, carbonate of RE and oxalate of RE.

[0013] In S1 of the above preparation method, the silica aerogel containing A is prepared by the following steps:

[0014] S11. Weighing silica aerogel and adding into ethanol solution containing A ions or colloidal nanoparticles of A, then stirring at 50°C to 75°C for 0.5h to 3h to thoroughly dissolve silica aerogel, obtaining mixed solution; where molar concentration of A ions or colloidal nanoparticles of A is in a range of 1.25×10^{-3} mol/L to1.5×10^{-5} mol/L; molar ratio of silica aerogel to A ions or colloidal nanoparticles of A is in a range of 3.3×10^{4} to 328:1.

[0015] S12. Stirring and ultrasonically treating the mixed solution, then drying the mixed solution at 60°C to 150°C to remove solvent, obtaining dried matter;

[0016] S13. Grinding the dried matter into powders, calcining the powders at 600°C to 1300°C for 0.5h to 3 h, cooling to room temperature to obtain silica aerogel containing A.

[0017] Preferably, in S11, aperture size of the silica aerogel is in a range of 20nm to 100 nm, porosity is in a range of 92% to 98%.

[0018] The silicate luminescent materials doped with metal nano particles of the invention show relatively high luminescent efficiency under excitation by electron beam, thus can be used in field emission light source devices.

[0019] In addition, the preparation method of the present invention is simple, non-polluting, high-quality, low-cost, and can be widely used in the manufacture of luminescent materials.

BRIEF DESCRIPTION OF THE DRAWINGS

[0020]

Fig. 1 is a flow diagram showing the preparation of silicate luminescent materials doped with metal nano particles;

Fig. 2 shows an emission spectrum of the silicate luminescent material doped with metal nano particles ($NaY_{0.9}SiO_4$: $0.1Tb^{3+}$, 0.003Ag) of Example 8, compared to the luminescent material without Ag nanoparticles ($NaY_{0.9}SiO_4$: $0.1Tb^{3+}$) of Comparative Example 1. Curve a is an emission spectrum of the silicate luminescent material doped with metal nano particles ($NaY_{0.9}SiO_4$: $0.1Tb^{3+}$, 0.003Ag) of Example 8, curve b is an emission spectrum of the luminescent material without Ag nanoparticles ($NaY_{0.9}SiO_4$: $0.1Tb^{3+}$) of Comparative Example 1.

DETAILED DESCRIPTION OF ILLUSTRATED EMBODIMENTS

[0021] In one embodiment, the silicate luminescent materials doped with metal nano particles is represented by the chemical formula of $MLn_{1-x}SiO_4$:xRE,yA; wherein M is one or two elements selected from Li, Na and K; Ln is one or two elements selected from Y, Sc, La and Lu; A is a metal nano particle selected from Ag, Au, Pt, Pd and Cu; RE is one or two ions selected from Eu, Gd, Tb, Tm, Sm, Ce and Dy; x and y are stoichiometric numbers, $0<x\leq0.1$; $0<y\leq0.005$. Preferably, x is in a range of $0.001\leq x\leq0.04$, y is in a range of $0.00003\leq y\leq0.003$.

[0022] As shown in Fig. 1, method for preparing the silicate luminescent materials doped with metal nano particles comprises the following steps:

S1, preparing silica aerogel containing A;

[0023] Firstly, silica aerogel was weighed and added into ethanol solution containing A ions or colloidal nanoparticles of A while stirring at 50°C to 75°C for 0.5h to 3 h, silica aerogel was thoroughly dissolved to form mixed solution, where molar concentration of A ions or colloidal nanoparticles of A was in a range of 1.25×10^{-3} mol/L to 1.5×10^{-5}mol/L, and molar ratio of silica aerogel to A ions or colloidal nanoparticles of A was in a range of 3.3×10^4 to 328:1.

[0024] Next, the mixed solution was stirred, ultrasonically treated, then dried at 60°C to 150°C to remove solvent and obtain dried matter;

[0025] At last, the dried matter was grinded into powders. The powders was calcined at 600°C to 1300°C for 0.5h to 3h, then cooled to room temperature to obtain silica aerogel containing A.

[0026] S2. According to the stoichiometric ratio of corresponding elements in the chemical formula of $MLn_{1-x}SiO_4$:xRE,yA, weighing compound containing M, compound containing Ln, compound containing RE and silica aerogel containing A ,then grinding and mixing to obtain mixed powders;

[0027] S3, in air or in reducing atmosphere, calcining the mixed powders obtained from S2 at a constant temperature ranged from 800°C to 1600°C for 1h to 10 h; then cooling to room temperature, taking out the calcined matter and grinding to obtain silicate luminescent material doped with metal nano particles represented by the chemical formula of $MLn_{1-X}SiO_4$:xRE,yA. During the calcination, the compound containing RE subjected to pyrolysis or reducing atmosphere is reduced to RE metal element.

[0028] M is one or two elements selected from Li, Na and K; Ln is one or two elements selected from Y, Sc, La and Lu; A is a metal nano particle selected from Ag, Au, Pt, Pd and Cu; RE is one or two ions selected from Eu, Gd, Tb, Tm, Sm, Ce and Dy; x and y are stoichiometric numbers, $0<x\leq0.1$; $0<y\leq0.005$.

[0029] In S1 of the above preparation method, aperture size of the silica aerogel is preferably in a range of 20-100 nm, porosity is preferably in a range of 92% to 98%.

[0030] In S2 of the above preparation method, the compound containing M is selected from oxide of M, nitrate of M, carbonate of M and oxalate of M; the compound containing Ln is selected from oxide of Ln, nitrate of Ln, carbonate of Ln and oxalate of Ln; the compound containing RE is selected from oxide of RE, nitrate of RE, carbonate of RE and oxalate of RE.

[0031] When weighing the compound containing M, an excess of compound containing M of 10 mol% (molar percent) is preferred.

[0032] In S3 of the above preparation method, the reducing atmosphere is preferably mixed gases of N_2 and H_2, the volume ratio of N_2 to H_2 is 95:5.

[0033] The silicate luminescent materials doped with metal nano particles of the invention show relatively high luminescent efficiency under excitation by electron beam, thus can be used in field emission light source devices.

[0034] The method for preparing silicate luminescent materials doped with metal nano particles of the present invention is simple, non-polluting, high-quality, low-cost, and can be widely used in the manufacture of luminescent materials.

[0035] Further description of the present invention will be illustrated, which combined with preferred embodiments and the drawings.

Example 1

$NaY_{0.999}SiO_4$: $0.001Tm^{3+}$, 0.00003Ag

[0036] Silica aerogel having an aperture size in a range of 20nm tolOOnm and porosity in a range of 92% to 98% was used. Firstly, 0.3 g of silica aerogel was weighed and added into 10 mL of ethanol solution containing 1.5×10^{-5}mol/L $AgNO_3$ while stirring at 50°C for 3 h, then ultrasonically treated for 10min and dried at 60°C. The dried matter was grinded into powders. The powders were pre-calcined at 600°C for 4h. 0.7150g of $Y_2(CO_3)_3$, 0.2543g of Na_2CO_3 (in excess of 20%), 0.0010g of $Tm_2(CO_3)_3$ and 0.2524g of silica aerogel containing Ag were weighed, grinded and mixed to obtain mixed powders. In air atmosphere, the mixed powders were calcined constantly at 800°C for 10 h. The obtained product was cooled to room temperature. The obtained silicate luminescent material doped with metal nano particles was represented by the chemical formula of $NaY_{0.999}SiO_4$:$0.001Tm^{3+}$, 0.00003Ag.

Example 2

$LiLa_{0.992}SiO_4$: $0.008Ce^{3+}$, 0.00007Au

[0037] Silica aerogel having an aperture size in a range of 20nm to 100nm and porosity in a range of 92% to 98% was used. Firstly, 0.4 g of silica aerogel was weighed and added into 20 mL of ethanol solution containing 2.345×10^{-5}mol/L colloidal nanoparticles of Au while stirring at 60°C for 2 h, then ultrasonically treated for 10min and dried at 80°C. The dried matter was grinded into powders. The powders were pre-calcined at 800°C for 2h. 0.6464g of La_2O_3, 0.1626g of Li_2CO_3 (in excess of 10%), 0.0055g of CeO_2 and 0.2525 g of silica aerogel containing Au were weighed, grinded and mixed to obtain mixed powders. In reducing atmosphere of mixed gases of N_2 and H_2 in a volume ratio of 95:5, the mixed powders were calcined constantly at 1450°C for 4 h. The obtained product was cooled to room temperature. The obtained silicate luminescent material doped with metal nano particles was represented by the chemical formula of $LiLa_{0.992}SiO_4$: $.008Ce^{3+}$, 0.00007Au.

Example 3

$KLu_{0.96}SiO_4$: $0.02Eu^{3+}$, 0.02Gd, 0.001Pt

[0038] Silica aerogel having an aperture size in a range of 20nm tolOOnm and porosity in a range of 92% to 98% was used. Firstly, 1.0 g of silica aerogel was weighed and added into 30mL of ethanol solution containing 5.43×10^{-4}mol/L colloidal nanoparticles of Pt while stirring at 70°C for 0.5 h, then ultrasonically treated for 10min and dried at 150°C. The dried matter was grinded into powders. The powders were pre-calcined at 1000°C for 4h. 0.4044g of KNO_3, 0.7640g of Lu_2O_3, 0.0137g of $Gd(NO_3)_3$, 0.0271g of $Eu(NO_3)_3$ and 0.2526g of silica aerogel containing Pt were weighed, grinded and mixed to obtain mixed powders. In air atmosphere, the mixed powders were calcined constantly at 1600°C for 1 h. The obtained product was cooled to room temperature. The obtained silicate luminescent material doped with metal nano particles was represented by the chemical formula of $KLu_{0.96}SiO_4$: $0.02Eu^{3+}$, 0.02Gd, 0.001Pt.

Example 4

$Na_{0.9}Li_{0.1}Sc_{2.2}Y_{0.792}SiO_4$: $0.008Dy^{3+}$, 0.0004Pd

[0039] Silica aerogel having an aperture size in a range of 20nm to 100nm and porosity in a range of 92% to 98% was used. Firstly, 0.28 g of silica aerogel was weighed and added into 15mL of ethanol solution containing 1.2266×10^{-4}mol/L colloidal nanoparticles of Pd while stirring at 60°C for 2 h, then ultrasonically treated for 10min and dried at 70°C. The dried matter was grinded into powders. The powders were pre-calcined at 900°C for 2h. 0.2003g of Na_2NO_3(in excess of 5%), 0.0163g of Li_2CO_3(in excess of 10%), 0.3577g of Y_2O_3, 0.0060g of Dy_2O_3 and 0.2524g of silica aerogel containing Pd were weighed, grinded and mixed to obtain mixed powders. In air atmosphere, the mixed powders were calcined constantly at 1350°C for 4 h. The obtained product was cooled to room temperature. The obtained silicate luminescent material doped with metal nano particles was represented by the chemical formula of $Na_{0.9}Li_{0.1}Sc_{0.2}Y_{0.792}SiO_4$: $0.008Dy^{3+}$, 0.0004Pd.

Example 5

$NaLu_{0.98}SiO_4$: $0.02Tb^{3+}$, 0.003Ag

[0040] Silica aerogel having an aperture size in a range of 20nm to 100nm and porosity in a range of 92% to 98% was used. Firstly, 0.35g of silica aerogel was weighed and added into 25 mL of ethanol solution containing 7.08×10^{-4}mol/L $AgNO_3$ while stirring at 65°C for 1.5 h, then ultrasonically treated for 10min and dried at 120°C. The dried matter was grinded into powders. The powders were pre-calcined at 1000°C for 2h. 0.2680g of $Na_2C_2O_4$, 1.2034g of $Lu_2(C_2O_4)_3$, 0.0233g of $Tb_2(C_2O_4)_3$ and 0.2523g of calcined silica aerogel were weighed, grinded and mixed to obtain mixed powders. In reducing atmosphere of mixed gases of N_2 and H_2 in a volume ratio of 95:5, the mixed powders were calcined constantly at 1400°C for 6 h. The obtained product was cooled to room temperature. The obtained silicate luminescent material doped with metal nano particles was represented by the chemical formula of $NaLu_{0.99}SiO_4$: $0.02Tb^{3+}$, 0.003Ag.

Example 6

$NaY_{0.95}SiO_4$: $0.05Sm^{3+}$, 0.005Ag

[0041] Silica aerogel having an aperture size in a range of 20nm to 100nm and porosity in a range of 92% to 98% was used. Firstly, 0.3 g of silica aerogel was weighed and added into 20mL of ethanol solution containing 1.25×10^{-3} mol/L $AgNO_3$ while stirring at 60°C for 2 h, then ultrasonically treated for 10min and dried at 100°C. The dried matter was grinded into powders. The powders were pre-calcined at 800°C for 2h. 0.3400g of $NaNO_3$, 0.4290g of Y_2O_3, 0.0349g of Sm_2O_3 and 0.2525g of calcined silica aerogel were weighed, grinded and mixed to obtain mixed powders. In air atmosphere, the mixed powders were calcined constantly at 1200°C for 6 h. The obtained product was cooled to room temperature. The obtained silicate luminescent material doped with metal nano particles was represented by the chemical formula of $NaY_{0.95}SiO_4$: 0.05 Sm^{3+}, 0.005Ag.

Example 7

$KLa_{0.99}SiO_5$: $0.01Ce^{3+}$, 0.0007Cu

[0042] Silica aerogel having an aperture size in a range of 20nm to 100nm and porosity in a range of 92% to 98% was used. Firstly, 0.4g of silica aerogel was weighed and added into 15 mL of ethanol solution containing 3.1266×10^{-4} mol/L colloidal nanoparticles of Cu while stirring at 60°C for 1.5 h, then ultrasonically treated for 10min and dried at 70°C. The dried matter was grinded into powders. The powders were pre-calcined at 800°C for 2h. 0.2764g of K_2CO_3, 0.6451g of La_2O_3, 0.0108g of $Ce_2(C_2O_4)_3$ and 0.2527g of silica aerogel containing Cu were weighed, grinded and mixed to obtain mixed powders. In reducing atmosphere of mixed gases of N_2 and H_2 in a volume ratio of 95:5, the mixed powders were calcined constantly at 1500°C for 4 h. The obtained product was cooled to room temperature. The obtained silicate luminescent material doped with metal nano particles was represented by the chemical formula of $KLa_{0.99}SiO_5$:$0.01Ce^{3+}$, 0.0007Cu.

Example 8

$NaY_{0.9}SiO_4$: $0.1Tb^{3+}$, 0.003Ag

[0043] Silica aerogel having an aperture size in a range of 20nm to 100nm and porosity in a range of 92% to 98% was used. Firstly, 0.35g of silica aerogel was weighed and added into 25 mL of ethanol solution containing 7.08×10^{-4}mol/L $AgNO_3$ while stirring at 65°C for 1.5 h, then ultrasonically treated for 10min and dried at 120°C. The dried matter was grinded into powders. The powders were pre-calcined at 800°C for 2h. 0.2332g of Na_2CO_3 (in excess of 10%), 0.4065g of Y_2O_3, 0.0748g of Tb_4O_7 and 0.2523g of calcined silica aerogel were weighed, grinded and mixed to obtain mixed powders. In reducing atmosphere of mixed gases of N_2 and H_2 in a volume ratio of 95:5, the mixed powders were calcined constantly at 1250°C for 6 h. The obtained product was cooled to room temperature. The obtained silicate luminescent material doped with metal nano particles was represented by the chemical formula of $NaY_{0.9}SiO_4$: $0.1Tb^{3+}$, 0.003Ag.

Comparative Example 1

$NaY_{0.9}SiO_4$:$0.1Tb^{3+}$

[0044] 0.2332g of Na_2CO_3 (in excess of 10 mol%), 0.4065g of Y_2O_3, 0.0748g of Tb_4O_7 and 0.2523g of silica aerogel were weighed, grinded and mixed to obtain mixed powders. In reducing atmosphere of mixed gases of N_2 and H_2 in a volume ratio of 95:5, the mixed powders were calcined constantly at 1250°C for 6 h. The obtained product was cooled to room temperature. The obtained luminescent material was represented by the chemical formula of $NaY_{0.9}SiO_4$: $0.1Tb^{3+}$.

[0045] Fig. 2 shows an emission spectrum of the silicate luminescent material doped with metal nano particles ($NaY_{0.9}SiO_4$: $0.1Tb^{3+}$, 0.003Ag) of Example 8, compared to the luminescent material without Ag nanoparticles ($NaY_{0.9}SiO_4$: $0.1Tb^{3+}$) of Comparative Example 1. Curve a is an emission spectrum of the silicate luminescent material doped with metal nano particles ($NaY_{0.9}SiO_4$: $0.1Tb^{3+}$, 0.003Ag) of Example 8, curve b is an emission spectrum of the luminescent material without Ag nanoparticles ($NaY_{0.9}SiO_4$: $0.1Tb^{3+}$) of Comparative Example 1.

[0046] As shown in Fig. 2, under excitation by cathode ray at 5KV, luminescent intensity of the silicate luminescent material doped with metal nano particles containing Ag nanoparticles ($NaY_{0.9}SiO_4$: $0.1Tb^{3+}$,0.003Ag) is increased by 30%, compared to the luminescent material without Ag nanoparticles.

[0047] While the present invention has been described with reference to particular embodiments, it will be understood that the embodiments are illustrative and that the invention scope is not so limited. Alternative embodiments of the present invention will become apparent to those having ordinary skill in the art to which the present invention pertains. Such alternate embodiments are considered to be encompassed within the spirit and scope of the present invention. Accordingly, the scope of the present invention is described by the appended claims and is supported by the foregoing description.

Claims

1. A silicate luminescent material doped with metal nano particles represented by the chemical formula:

   $MLn_{1-x}SiO_4$:xRE,yA; wherein M is one or two elements selected from Li, Na and K; Ln is one or two elements selected from Y, Sc, La and Lu; A is a metal nano particle selected from Ag, Au, Pt, Pd and Cu; RE is one or two ions selected from Eu, Gd, Tb, Tm, Sm, Ce and Dy; x and y are stoichiometric numbers, $0<x\leq0.1$; $0<y\leq0.005$.

2. The silicate luminescent material doped with metal nano particles according to claim 1, wherein x is in a range of $0.001\leq x\leq0.04$, y is in a range of $0.00003\leq y\leq0.003$.

3. A method for preparing silicate luminescent material doped with metal nano particles, comprising:

S1. according to the stoichiometric ratio of corresponding elements in the chemical formula of $MLn_{1-x}SiO_4$:xRE,yA,weighing compound containing M, compound containing Ln, compound containing RE and silica aerogel containing A , then grinding and mixing to obtain mixed powders;

S2, in air or in reducing atmosphere, calcining the mixed powders obtained from S1 at a constant temperature ranged from 800°C to 1600°C for 1hto 10h; then cooling to room temperature, taking out the calcined matter and grinding to obtain silicate luminescent material doped with metal nano particles represented by the chemical formula of $MLn_{1-x}SiO_4$:xRE,yA;

wherein M is one or two elements selected from Li, Na and K; Ln is one or two elements selected from Y, Sc, La and Lu; A is a metal nano particle selected from Ag, Au, Pt, Pd and Cu; RE is one or two ions selected from Eu, Gd, Tb, Tm, Sm, Ce and Dy; x and y are stoichiometric numbers, $0<x\leq0.1$; $0<y\leq0.005$.

4. The method for preparing silicate luminescent material doped with metal nano particles according to claim 3, wherein in S1, the compound containing M is selected from oxide of M, nitrate of M, carbonate of M and oxalate of M; the compound containing Ln is selected from oxide of Ln, nitrate of Ln, carbonate of Ln and oxalate of Ln; the compound containing RE is selected from oxide of RE, nitrate of RE, carbonate of RE and oxalate of RE.

5. The method for preparing silicate luminescent material doped with metal nano particles according to claim 2, wherein in S1, the silica aerogel containing A is prepared by the following steps:

S11. weighing silica aerogel and adding into ethanol solution containing A ions or colloidal nanoparticles of A, then stirring at 50°C to 75°C for 0.5h to 3h to thoroughly dissolve silica aerogel, obtaining mixed solution; wherein molar concentration of A ions or colloidal nanoparticles of A is in a range of 1.25×10^{-3} mol/L to 1.5×10^{-5}mol/L;

S 12. stirring and ultrasonically treating the mixed solution, then drying the mixed solution at 60°C to 150°C to remove solvent, obtaining dried matter;

S13. grinding the dried matter into powders, calcining the powders at 600°C to 1300°C for 0.5h to 3 h, cooling to room temperature to obtain silica aerogel containing A.

6. The method for preparing silicate luminescent material doped with metal nano particles according to claim 5, wherein in S11, molar ratio of silica aerogel to A ions or colloidal nanoparticles of A is in a range of $3.3x10^4$to 328:1.

7. The method for preparing silicate luminescent material doped with metal nano particles according to claim 6, wherein in S11, aperture size of the silica aerogel is in a range of 20nm to 100 nm, porosity is in a range of 92% to 98%.

8. The method for preparing silicate luminescent material doped with metal nano particles according to claim 3, wherein in S2, the reducing atmosphere is mixed gases of N_2 and H_2 in a volume ratio of 95:5.

9. The method for preparing silicate luminescent material doped with metal nano particles according to any one of the claims from 3 to 8, wherein x is in a range of $0.001\leq x\leq0.04$, y is in a range of $0.00003\leq y\leq0.003$.

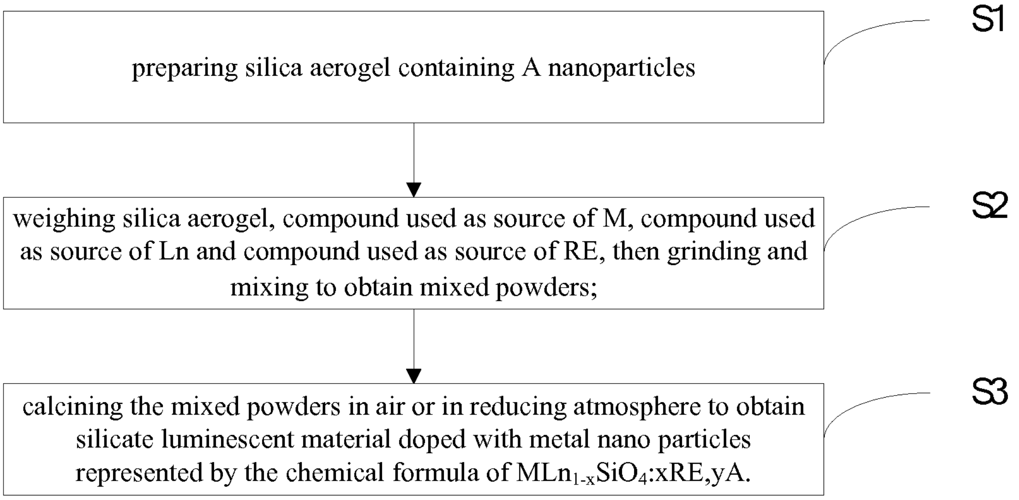
preparing silica aerogel containing A nanoparticles
S1
weighing silica aerogel, compound used as source of M, compound used as source of Ln and compound used as source of RE, then grinding and mixing to obtain mixed powders;
S2
calcining the mixed powders in air or in reducing atmosphere to obtain silicate luminescent material doped with metal nano particles represented by the chemical formula of $MLn_{1-x}SiO_4$:xRE,yA.
S3

Fig. 1

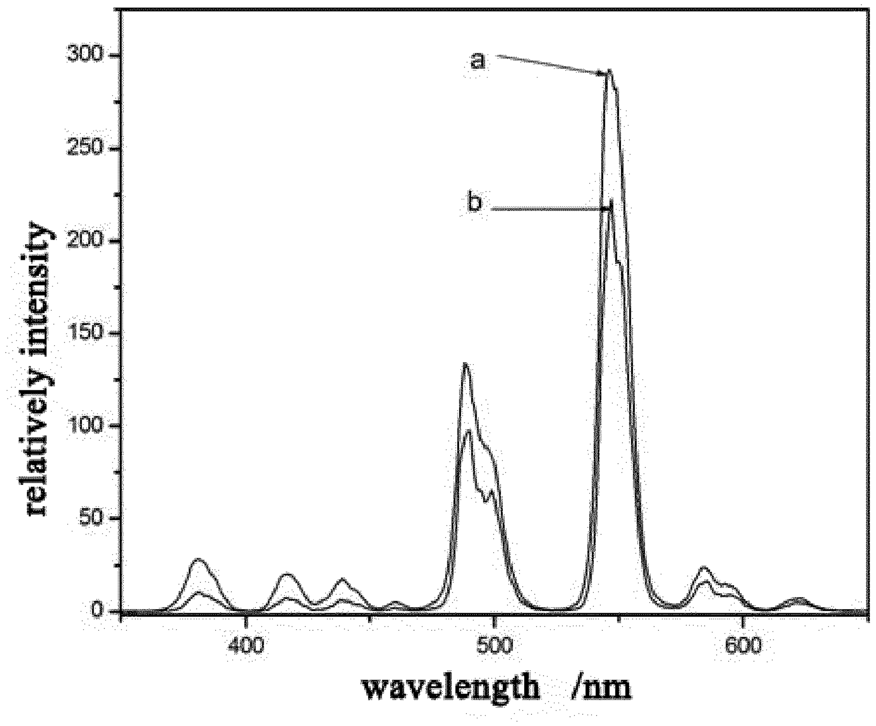
a
b
300
250
200
150
100
50
0
400
500
600
relatively intensity
wavelength /nm

Fig. 2

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2012/073245

A. CLASSIFICATION OF SUBJECT MATTER

C09K 11/79 (2006.01) i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
WPI, EPODOC, CNKI, CPRS, CA: OCEAN'S KING LIGHTING; WANG, Rong; CHEN, Guitang; LIU, Jun; ZHOU, Mingjie; lithium, natrium, kalium, oxygen, silicium, yttrium, scandium, lanthanum, lutetium, Li, Na, K, O, Si, Y, Sc, La, Lu, Ag, Au, Pt, Pd, Cu, core, shell, coat, nano

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 102382646 A (OCEAN'S KING LIGHTING SCIENCE & TECHNOLOGY CO., LTD. et al.), 21 March 2012 (21.03.2012), see claims 1-10, and embodiments	1-9
A	CN 101033398 A (LANZHOU UNIVERSITY), 12 September 2007 (12.09.2007), see claims 1-5	1-9

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search 17 December 2012 (17.12.2012)	Date of mailing of the international search report **03 January 2013 (03.01.2013)**
Name and mailing address of the ISA/CN: State Intellectual Property Office of the P. R. China No. 6, Xitucheng Road, Jimenqiao Haidian District, Beijing 100088, China Facsimile No.: (86-10) 62019451	Authorized officer **DI, Yanxin** Telephone No.: (86-10) **62086319**

Form PCT/ISA/210 (second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CN2012/073245

Patent Documents referred in the Report	Publication Date	Patent Family	Publication Date
CN 102382646 A	21.03.2012	None	
CN 101033398 A	12.09.2007	CN 100560687 C	18.11.2009

Form PCT/ISA/210 (patent family annex) (July 2009)